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EUROPEAN PATENT APPLICATION

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(54) **Process for producing high-purity aluminum.**

(57) To obtain a high-purity aluminum fraction from aluminum containing both eutectic impurities and peritectic impurities, the contents of these impurities in the original aluminum are reduced by melting the original aluminum to obtain molten aluminum (1), adding boron to the molten aluminum (1), and rotating a cooling body (3) as immersed in the boron-containing molten aluminum (1) while introducing a cooling fluid to the interior of the body (3) to crystallize high-purity aluminum on the surface of the body (3). The peritectic impurities react with the boron to form metallic borides, which are centrifugally forced away from the cooling body (3) by the rotation of the latter without being incorporated into the aluminum crystallized on the surface of the body (3). The eutectic impurities are removed from the crystallized aluminum on the surface based on the principle of segregation.

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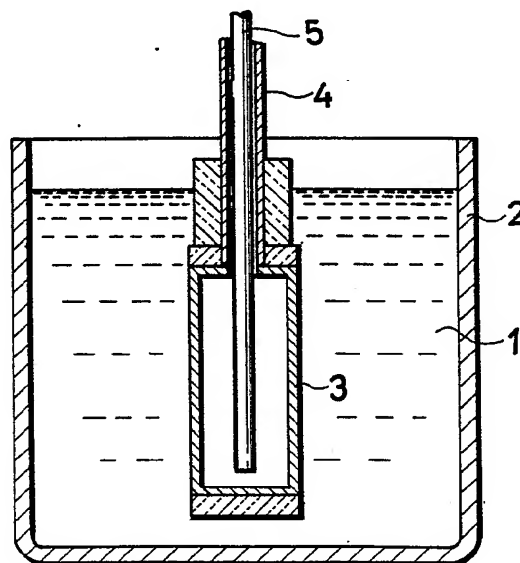
PROCESS FOR PRODUCING HIGH-PURITY ALUMINUM

The present invention relates to a process for producing aluminum having high purity, and more particularly to a process for treating aluminum containing

5 eutectic impurities, such as Fe, Si Cu and Mg, and also peritectic impurities, such as Ti, V and Zr, by resorting to segregation solidification to obtain aluminum having lower contents of eutectic impurities and peritectic impurities and a higher purity than the original aluminum.

10 Throughout the specification and the appended claims, the term "eutectic impurities" refers to the impurities which undergo a eutectic reaction with aluminum, and the term "peritectic impurities" refers to the impurities which undergo a peritectic reaction with

15 aluminum.

When aluminum containing both eutectic

impurities and peritectic impurities is melted and then

subjected to equilibrium solidification, the impurity

concentration Cs of the resulting solid aluminum fraction

is theoretically represented by the following equation.

5 $$Cs = ko\, Co(1 - fs)^{ko-1} \qquad (1)$$

wherein ko is the distribution coefficient, Co is the

impurity concentration of the original aluminum, and fs

is the ratio of the solid fraction. The above equation

shows that when the distribution coefficient ko is

10   smaller than 1, the impurity concentration Cs of the solid

fraction is smaller than the impurity concentration Co of

the original aluminum and that when the distribution

coefficient ko is larger than 1, the impurity concentra-

tion Cs of the solid fraction is greater than the impurity

15   concentration Co of the original aluminum. Since

eutectic impurities are smaller than 1 in distribution

coefficient, the solid fraction prepared from aluminum

containing such impurities is lower than the original

aluminum in impurity concentration. Accordingly when the

20   original aluminum is melted and thereafter solidified,

high-purity aluminum can be obtained advantageously by

selectively separating off the resulting pro-eutectic

aluminum. Segregation solidification processes based

on this principle are already known as disclosed, for

25   example, in U.S. Patents No. 3,671,229, No. 3,211,547,

etc. However, peritectic impurities are larger than 1
in distribution coefficient, so that aluminum containing
such impurities affords a solid fraction which is higher
than the original aluminum in impurity concentration.

5   Accordingly when aluminum containing both eutectic
impurities and peritectic impurities is treated by the
conventional segregation solidification process to obtain
pro-eutectic aluminum selectively, the resulting solid
fraction is lower in the concentration of eutectic

10   impurities but higher in the concentration of peritectic
impurities than the original aluminum.

     Aluminum containing both eutectic impurities
and peritectic impurities is therefore usually treated
by adding boron to a melt of such aluminum in a container

15   to cause the boron to react with Ti, V, Zr and like
peritectice impurities and form insoluble metallic borides
such as TiB_2, VB_2, ZrB_2, etc., allowing the mixture to
stand for a long period of time, for example, for more
than 1 hour to settle the metallic borides, placing the

20   molten aluminum into another container as separated from
the borides, and selectively drawing off pro-eutectic
aluminum only from the container. This process is
inefficient since the settlement of the metallic borides
requires much time.

The object of the present invention is to
overcome the above drawback and to provide a process for
treating aluminum containing both eutectic impurities
and peritectic impurities by segregation solidification
5   to remove the impurities of both types from the aluminum
to the greatest possible extent and obtain aluminum
having a higher purity than the original aluminum.

The process of this invention for producing
high-purity aluminum comprises the steps of melting
10   aluminum containing both eutectic impurities and peritec-
tic impurities to obtain molten aluminum, adding boron
to the molten aluminum, immersing a cooling body into
the boron-containing molten aluminum, and rotating the
cooling body while introducing a cooling fluid to the
15   interior of the cooling body to crystallize high-purity
aluminum on the surface of the body.

With this process, the boron reacts with the
peritectic impurities to form metallic borides, which
are moved away from the cooling body and dispersed
20   throughout the liquid phase by the centrifugal force
produced by the rotation of the cooling body. Accordingly
an aluminum fraction having lower contents of eutectic
impurities and peritectic impurities than the original
aluminum can be crystallized on the surface of the cool-
25   ing body, giving high-purity aluminum. Unlike the

conventional process, the present process does not
involve the necessity of settling the metallic borides
and therefore can be practiced within a shortened period
of time.

5   When aluminum containing Fe, Si, Cu, Mg and
like eutectic impurities, and Ti, V, Zr and like peritec-
tic impurities is melted, and boron is added to the
resulting melt, the boron reacts with the Ti, V, Zr and
like peritectic impurities to form TiB_2, VB_2, ZrB_2 and
10   like insoluble metallic borides. The amount of boron
to be used, which is determined in accordance with the
Ti, V, Zr and like contents of the aluminum to be puri-
fied, is a predetermined amount larger than the amount
needed for forming the above metallic borides. The boron
15   may be added in the form of an Al-B master alloy or $NaBF_4$
or like salt. Since boron undergoes a eutectic reaction
with aluminum, the excess of boron other than the quanti-
ty of boron forming the metallic borides with the
peritectic impurities is removed in the same manner as
20   the eutectic impurities as will be described below.
        When a cooling body is rotated as immersed in
the boron-containing molten aluminum while introducing
a cooling fluid to the interior of the body, high-purity
aluminum crystallizes on the surface of the cooling body.
25   Stated more specifically, when aluminum containing

eutectic impurities is melted and subjected to equilibrium

solidification, pro-eutectic aluminum of high purity

separates out first. The ratio between the eutectic

impurity concentration of the solid phase and the eutectic

5   impurity concentration of the liquid phase at this time

is represented by the distribution coefficient ko. During

actual solidification, however, the eutectic impurities

are released into the liquid phase to form a layer of

concentrated eutectic impurities in the liquid phase in

10   the vicinity of the liquid-solid interface or boundary,

so that the impurity concentration of the solid phase

is governed by the effective distribution coefficient k_E

considering the thickness of the concentrated layer (here-

inafter referred to as "diffusion layer"). Thus the

15   impurity concentration Cs of the solid phase is repre-

sented by Equation (1) wherein k_E is used in place of ko,

i.e. by the following equation.

$$Cs = k_E Co(1 - fs)^{k_E - 1} \qquad (2)$$

It is already known that the effective

20   distribution coefficient k_E is represented by the follow-

ing equation.

$$k_E = \frac{ko}{ko + (1 - ko) \exp\left(-\frac{R\delta}{D}\right)}$$

wherein R is the solidification velocity, δ is the thick-

ness of the diffusion layer formed in the vicinity of the

liquid-solid interface, and D is the diffusion coeffi-
cient of the eutectic impurities in the liquid phase. It
therefore follows that the effective distribution
coefficient k_E can be made approximate to the distribu-

5   tion coefficient ko effectively by decreasing the solid-
ification velociy R and the thickness of the diffusion
layer, δ , and increasing the diffusion coefficient D. Of
these variables, the diffusion coefficient D is considered
to be a constant which is dependent on the kind of the

10 eutectic impurity generally when the liquid phase has a
constant temperature, so that it is difficult to vary
the diffusion coefficient D. It is therefore effective
to decrease the solidification velocity R and the thick-
ness of the diffusion layer, δ , for making the effective

15 distribution coefficient k_E approximate to the distribu-
tion coefficient ko.

When the eutectic impurities are released into
the liquid phase at the liquid-solid interface, dendrites
are formed at the interface. It is well known that when

20 such dendrites grow, the impurities released into the
liquid phase in the vicinity of the liquid-solid
interface are trapped, as they are or in the form of
eutectic crystals of several microns, in the interstices
of the dendrites. This phenomenon is undesirable for

25 the purification of aluminum. The growth of dendrites

occurs when the actual temperature of the liquid phase
is lower than the temperature of the equilibrium
liquidus in the vicinity of the liquid-solid
interface. This phenomenon is referred to as constitu-
tional undercooling. The growth of dendrites is promoted
when the difference (degree of undercooling) ΔT between
the temperature of the equilibrium liquidus and the
actual liquid phase temperature increases. Accordingly
the growth of dendrites can be inhibited effectively by ·
decreasing the difference ΔT. To decrease ΔT, there is
the need to increase the temperature gradient of the
melt in the vicinity of the liquid-solid interface.
Ideally, the temperature gradient should be smaller than
the temperature gradient of the equilibrium liquidus
to make ΔT smaller than 0.

   The foregoing leads to the following three
requirements for purifying aluminum with improved
efficiency.

(a) To decrease the solidification velocity to the great-
    est possible extent.

(b) To decrease to the greatest possible extent the thick-
    ness of the diffusion layer in the vicinity of the
    liquid-solid interface.

(c) To increase to the greatest possible extent the
    temperature gradient involved in the liquid phase in

the vicinity of the interface.

When a cooling body is immersed in molten aluminum containing boron, aluminum having a high purity starts to crystallize on the surface of the cooling body.

5 The solidification proceeds while the eutectic impurities are being released into the liquid phase in the vicinity of the liquid-solid interface to form a diffusion layer. The solidification velocity at this time is governed by the difference between the amount of heat removed from

10 the molten aluminum by the cooling body and the amount of heat supplied to the molten aluminum by heating from outside. Thus the velocity is controlled by these amounts of heat. Further when an increased amount of heat is supplied to the molten aluminum by external heating, with

15 an increased amount of heat removed from the melt by cooling with the cooling body, greater transfer of heat occurs between the cooling body and the melt, consequently giving a greater temperature gradient to the liquid phase in the vicinity of the liquid-solid interface. The

20 greater the temperature gradient, the more effectively is the formation of dendrites inhibited at the interface to prevent the eutectic impurities from being arrested by the dendrites. Ideally, the temperature gradient involved in the liquid phase in the vicinity of the

25 liquid-solid interface should be greater than that of

the equilibrium liquidus. The formation of dendrites
can then be inhibited completely. From the viewpoint of
industrial productivity, however, it is desirable to
effect suitable transfer of heat from the molten aluminum
5   to the cooling body by advantageously controlling the
external heating and cooling with the cooling body.
Further when the cooling body is rotated, the solid phase
and the liquid phase move relative to each other, whereby
the diffusion layer formed near the liquid-solid inter-
10   face is effectively agitated and mixed with the other
major portion of the liquid phase to disperse the impuri-
ites in the diffusion layer throughout the entire liquid
phase, consequently greatly reducing the thickness of the
diffusion layer. At the same time, the boundary layer
15   in the temperature distribution of the liquid phase is
made thinner, consequently permitting the liquid phase
to have an increased temperature gradient in the vicinity
of the liquid-solid interface. The thicknesses of the
diffusion layer and the boundary layer in the temperature
20   distribution can be decreased by increasing the speed of
rotation of the cooling body, in other words, the speed
of the relative movement between the cooling body and
the molten aluminum. However, if the speed is exceedingly
high, the aluminum crystallized on the surface of the
25   cooling body is likely to separate off easily due to the

increase of the centrifugal force, resulting in reduced productivity. Preferably the speed of the relative movement is within the range of 400 to 8000 mm/sec because if the speed is lower than 400 mm/sec, the diffusion layer

5 will not be made thinner effectively, whereas if it is higher than 8000 mm/sec, the increased centrifugal force will lead to reduced productivity. When the cooling body is in the form of a cylinder having a diameter of 160 mm, for example, the body can be rotated at 50 to 1000

10 r.p.m., but especially good results are obtained at 200 to 700 r.p.m. Further improved results can be achieved if the direction of rotation of the body is reversed with suitable frequency. Since boron undergoes a eutectic reaction with aluminum, the excess of boron other

15 than the amount of boron forming metallic borides with the peritectic impurities is removed in the same manner as Fe, Si, Cu, Mg and like eutectic impurities.

The high-purity aluminum deposited on the surface of the cooling body on crystallization is

20 recovered, for example, by melting the high-purity fraction or by scraping off the fraction.

The present invention further provides a process comprising the steps of melting aluminum containing both eutectic impurities and peritectic impurities

25 to obtain molten aluminum, placing the molten aluminum

into a holding container, adding boron to the molten

aluminum within the container, and rotating a cooling

body as immersed in the boron-containing molten aluminum

within the container while introducing a cooling fluid

5   to the interior of the cooling body to crystallize high-

purity aluminum on the surface of the body.

In the above process, the cooling body is

immersed into the molten aluminum preferably after caus-

ing the boron to react with the peritectic impurities by

10   allowing the molten aluminum to stand for at least about

5 minutes following the addition of the boron. To

promote the reaction, it is desirable to agitate the

boron-containing molten aluminum.

The present invention further provides a

15   process comprising melting aluminum containing both eutec-

tic impurities and peritectic impurities to obtain molten

aluminum, placing the molten aluminum into a boron adding

vessel, causing the molten aluminum placed into the vessel

to be forwarded to a plurality of purifying tanks from

20   tank to tank and to be discharged from the last tank,

adding boron to the molten aluminum within the vessel, and

rotating a cooling body as immersed in the boron-contain-

ing molten aluminum within each of the tanks while

introducing a cooling fluid to the interior of the cooling

25   body to crystallize high-purity aluminum on the surface

of the body. This process affords a large quantity of high-purity aluminum efficiently by a continuous operation.

The boron adding vessel and the purifying tanks are prepared, for example, by dividing a single
5 large container into a plurality of sections by partitions each having a communication opening, one of the sections serving as the vessel and the other sections as the tanks. Alternatively a plurality of crucibles may be arranged in a row, with each crucible communicating with another
10 through a trough, such that one of the crucibles serves as the boron adding vessel and the others as the purifying tanks.

Boron is placed into the vessel continuously so that the molten aluminum therein has a constant boron
15 concentration during operation. Preferably the molten aluminum in the vessel is agitated to accelerate the reaction between the boron and the peritectic impurities. The cooling body is immersed in the molten aluminum in each of the purifying tanks. The supply of the melt to
20 the purifying tanks is so adjusted that the amount of the melt in each tank remains unchanged during operation. To allow equal amounts of aluminum to crystalize on the surfaces of the cooling bodies in the purifying tanks, the temperature of the molten aluminum, the speed of
25 rotation and cooling capacity of the cooling bodies are

suitably controlled. The eutectic impurity concentration of the molten aluminum in the purifying tanks progressively increases from tank to tank away from the melting furnace. The average impurity concentration of all the aluminum

5   fractions obtained is variable by varying the ratio of the total amount of aluminum fractions recovered by the cooling bodies to the total amount of the melt supplied. (The ratio will be referred to as "recovery ratio".) The smaller the recovery ratio, the lower is the average

10   impurity concentration. Accordingly aluminum having a constant purity which is higher than the purity of the original aluminum supplied from the melting furnace for purification can be obtained by rendering the recovery ratio constant. Further at the same recovery ratio, the

15   purity of the aluminum obtained increases with the increase in the number of purifying tanks.

The invention will be described below in greater detail with reference to the accompanying drawings.

Fig. 1 is a view in vertical section showing a

20   first embodiment of apparatus for producing high-purity aluminum by the process of the invention;

Fig. 2 is a view in vertical section showing a second embodiment of apparatus for producing high-purity aluminum by the process of the invention;

Fig. 3 is an enlarged fragmentary view of
Fig. 2 partly broken away;

Fig. 4 is a graph showing the result achieved
when the process of the invention was practiced with use
5 of the second apparatus, the graph showing the relation
of the amount of aluminum (yield) crystallized on the
peripheral surface of a cooling body in each purifying
tank to the ratio of the eutectic impurity concentration
of the molten aluminum in the tank to the eutectic
10 impurity concentration of the aluminum to be purified; and

Fig. 5 is a graph showing the relation between
the number of purifying tanks and the ratio of the eutec-
tic impurity concentration of the aluminum obtained to
that of the aluminum to be purified, the relation being
15 determined by practicing the process of the invention with
use of the second apparatus.

With reference to a first apparatus for
practicing the process of the invention, the aluminum to
be purifyied and containing both eutectic impurities and
20 peritectic impurities is melted and placed in a holding
container comprising a crucible 2. The molten aluminum 1
is heated and maintained at a temperature exceeding the
solidifying temperature thereof. Boron is added to the
molten aluminum 1, and the mixture is agitated for at

least 5 minutes. A cooling body 3 is thereafter immersed

in the melt 1. The cooling body 3 is in the form of a

hollow cylinder having closed opposite ends and is movable

upward and downward. A hollow rotary shaft 4 extends

5   upward from the upper end of the body 3. The interior of

the hollow rotary shaft 4 is in communication with the

interior of the cooling body 3. A cooling fluid supply

pipe 5 extends through the shaft 4 into the cooling body 3.

The rotary shaft 4 is provided at its upper end with a

10   drive device (not shown), by which the shaft 4 and the

body 3 are rotated. The top and bottom of the body 3 and

the portion of the shaft 4 to be exposed to the melt 1

are preferably covered with a heat insulating material 6

so as not to permit aluminum to crystallize on these

15   portions. This is due to the reason that since the effect

produced by the rotation of the body 3 is lower at these

portions than at the periphery of the body 3, aluminum,

if crystallizing on these portion, will have a lower purity

than the aluminum crystallizing on the periphery. The

20   cooling body 3 is rotated while introducing a cooling

fluid, such as air, nitrogen or argon, from the pipe 5

into the body 3 at an adjusted rate so that the portion

of the melt 1 around the body 3 will be cooled slowly.

Pro-eutectic aluminum having a high purity then starts to

25   crystallize on the periphery of the body 3. After a

specified quantity of aluminum has been deposited on
the body 3, the body is raised and withdrawn from the
melt 1, and the high-purity aluminum obtained is collected.

The above procedure is repeated in aluminum
5   melts of progressively higher purities to obtain aluminum
of greatly improved purity.

With reference to Figs. 2 and 3 showing a second
apparatus, there is provided a melting furnace 11 for
melting the aluminum to be purified and containing both
10   eutectic impurities and peritectic impurities. A large
container 12 in the form of a horizontally elongated
rectangular parallelepiped is disposed on the right side
of the furnace 11. The container 12 is divided into five
sections by partitions 13. Of these sections, the one
15   at the left end serves as a boron adding vessel 14, and
the other four sections serve as purifying tanks 15 to
18. The partitions 13 are formed at their upper ends
with openings 19 through which the vessel 14 and the tanks
15 to 18 communicate with one another. The vessel 14
20   has an agitator 20 in its interior. The agitator 20
comprises a vertical rotary shaft 21, a propeller-shaped
stirring blade 22 attached to the lower end of the shaft
21 and unillustrated drive means. Each of the tanks 15
to 18 is provided with a cooling body 23 which is movable
25   upward and downward. The body 23 is in the form of a

downwardly tapered hollow cylinder having closed opposite

ends and a hollow rotary shaft 24 extending upward from

its top.   The interior of the shaft 24 is in communication

with the interior of the body 23.   A cooling fluid supply

5   pipe 26 extending through the shaft 24 into the body 23

has a large number of cooling fluid outlets 25 in its

peripheral wall.   The upper end peripheral portion and

the bottom of the cooling body 23 are covered with heat

insulating members 27 and 28 respectively.

10   Before molten aluminum 30 is placed into the

tanks 14 to 18, the cooling bodies 23 are held raised

as indicated in broken lines in Fig. 2.

The aluminum to be purified and containing both

eutectic impurities and peritectic impurities is melted

15   in the furnace 11, and the molten aluminum 30 is first

placed into the vessel 14, in which boron is added to the

melt 30.   The mixture is agitated by the agitator 20.   In

this vessel 14, the boron reacts with Ti, V, Zr and

like peritectic impurities, forming TiB_2, VB_2, ZrB_2 and

20   like insoluble metallic borides.   The molten aluminum

containing the eutectic impurities, insoluble metallic

borides and an excess of boron flows into the tanks 15 to

18 through the communication openings 19.   After a

predetermined amount of molten aluminum 30 has been placed

25   into each of the tanks 15 to 18, the cooling body 23 is

lowered and immersed in the melt, with the upper heat

insulating member 27 in contact with the melt 30 and with

the lower edge of the member 27 located within the mass

of the melt 30 at such a position that it will not be

5   influenced by the temperature of the atmosphere. The

upper end peripheral portion of the body 23 is covered

with the insulating member 27, and the body is so immersed

in the melt 30 that the member 27 is in contact with the

surface of the melt 30 with its lower edge at a position

10   where it is free of the influence of the atmospheric

temperature, for the following reason. The portion of

the melt 30 near the surface thereof exposed to the

atmosphere has a lower temperature than the other portion

by being affected by the atmospheric temperature and is

15   therefore liable to solidify on the periphery of the body

23 in the vicinity of the above surface. Consequently

aluminum solidifies at a higher velocity at this portion

than at the other portion of the periphery of the body 23,

with the result that the aluminum fraction deposited on

20   this portion has a lower purity than the fraction on the

other portion and becomes mixed with the latter fraction

when the purified aluminum is removed from the outer

periphery of the body 23 for collection after the comple-

tion of the purifying operation. This results in a

25   reduced overall purifying efficiency. Further the bottom

of the cooling body 23 is covered with the heat insulat-
ing member 28 because the rotation of the body 23 will
not produce a satisfactory effect at the bottom, such that
the aluminum fraction deposited on the bottom of the body

5   23 has a lower purity than the other fraction on the
periphery of the body.

The cooling body 23 in this state is rotated
while injecting a cooling fluid into the body through
the inlets 25 of the supply pipe 26. This causes aluminum

10   to crystallize on the portion of the peripheral surface
of the body 23 which is immersed in the melt 30 and which
is not covered with the insulating members 27 and 28.
Since the metallic borides contained in the melt 30 are
centrifugally forced away from the body 23 by the rotation

15   thereof, the aluminum deposited on the periphery of the
body 23 is free from such borides. The excess of boron,
which forms a eutectic with aluminum, is removed like the
eutectic impurities. The temperature of the molten
aluminum, and the speed of rotation and cooling capacity

20   of the bodies 23 are so controlled that equal amounts of
aluminum will be deposited on the peripheral surfaces of
the bodies in the purifying tanks 15 to 18, while the
supply and discharge of molten aluminum are controlled
so that the tanks 15 to 18 contain equal amounts of melt,

25   with the recovery ratio maintained at a constant value.

Assuming that the molten aluminum supplied from the furnace 11 has a eutectic impurity concentration of $C0$ and that the melt portions contained in the tanks 15 to 18 have eutectic impurity concentrations of $C1$, $C2$, $C3$ and

5    $C4$ respectively, $C0 < C1 < C2 < C3 < C4$. Further the ratio of the eutectic impurity concentration of the melt in each of the tanks 15 to 18 to $C0$, namely the ratio $CN/C0$ where N is 1 to 4, increases with the start of the operation but levels off when the amount of aluminum

10   deposit on the periphery of the cooling body 23 reaches a value as shown in Fig. 4. At this time, the total amount of eutectic impurities, Ms, in the aluminum recovered by the cooling bodies 23 plus the amount of eutectic impurities, Mn, in the molten aluminum discharged

15   from the tank 18 at the right end is always equal to the amount of eutectic impurities, Mo, in the aluminum to be purified. Accordingly, if the eutectic impurity concentration and the effective distribution coefficient of the eutectic impurities of the molten aluminum in each

20   of the tanks 15 to 18 are known, high-purity aluminum can be obtained with the desired eutectic impurity concentration when the recovery ratio is set to a definite value. The smaller the recovery ratio, the lower is the eutectic impurity concentration of the molten aluminum in each

25   purifying tank, so that the cast block obtained has a

higher purity.   The purity of the aluminum to be obtained

can be determined by varying the recovery ratio.   At a

definite recovery ratio, the eutectic impurity concentra-

tion of the molten aluminum discharged increases with the

5   increase of the number of purifying tanks as seen in

Fig. 5, enabling the cooling bodies 23 to afford aluminum

of higher purity.

When the same operation as above is repeated

again with use of the aluminum collected by the cooling

10   bodies 23, aluminum having a higher purity can be

obtained.   Since the molten aluminum resulting from the

second operation usually has a higher purity than the

original aluminum used as the material for the first

operation, the molten aluminum, if returned to the

15   furnace 11, gives an improved yield.

Example 1

The apparatus shown in Fig. 1 was used.   Aluminum

containing 0.08 wt. % of Fe, 0.05 wt. % of Si, 0.0015 wt.

% of Ti, 0.004 wt. % of V and 0.0005 wt. % of Zr was

20   melted, and the molten aluminum 1 was maintained at 670°

C in the crucible 2.   Al-3 wt. % B master alloy was added

to the aluminum 1 so that the resulting melt had a boron

concentration of 0.004 wt. %.   The mixture was agitated

for 5 minutes after the addition of the alloy.   The

25   cooling body 3, 100 mm in outside diameter, was immersed

in the melt 1 and rotated at a speed of 400 r.p.m. while

supplying air into the body 3 through the supply pipe 5.

The body 3 was rotated for 10 minutes and thereafter

withdrawn from the melt 1 by raising. The body 3 was

5   found to have aluminum deposited on its periphery in an

amount of 20 wt. % based on the amount of the original

aluminum. When checked for impurity concentration, the

cast aluminum block was found to contain 0.008 wt. % of

Fe, 0.012 wt. % of Si, less than 0.0001 wt. % of Ti,

10   0.0002 wt. % of V, less than 0.0002 wt. % of Zr and

0.0003 wt. % of B.

Example 2

The apparatus shown in Fig. 1 was also used.

Aluminum containing 0.08 wt. % of Fe, 0.05 wt. % of Si,

15   0.002 wt. % of Ti, 0.008 wt. % of V and 0.001 wt. % of Zr

was melted, and the molten aluminum 1 was maintained at

670° C in the crucible 2. Al-3 wt. % B master alloy was

added to the aluminum 1 so that the resulting melt had a

boron concentration of 0.008 wt. %. The mixture was

20   agitated for 5 minutes after the addition of the alloy.

The cooling body 3, 200 mm in outside diameter, was then

immersed in the melt 1 and rotated at a speed of 650 r.p.m.

while supplying air into the body through the pipe 5.

After rotating the body 3 for 10 minutes, the body 3 was

25   withdrawn from the melt. The body 3 was found to have

aluminum deposited on its periphery in an amount of 20% based on the amount of the original aluminum. When checked for impurity concentration, the cast aluminum block was found to contain 0.005 wt. % of Fe, 0.009 wt. % of Si,

5 less than 0.0001 wt. % of Ti and V, less than 0.0002 wt. % of Zr and 0.0005 wt. % of B.

Example 3

The apparatus shown in Figs. 2 and 3 was used. The cooling bodies 23 were held raised. Aluminum contain-

10 ing 0.08 wt. % of Fe, 0.05 wt. % of Si, 0.002 wt. % of Ti and 0.008 wt. % of V was melted in the furnace 11, and the molten aluminum was supplied to the boron adding vessel 14. Boron was added to the aluminum so that the resulting melt had a boron concentration of 0.008 wt. %

15 at all times. The mixture was agitated by the agitator 20. When the purifying tanks 15 to 18 were filled with the molten aluminum flowing through the communication openings 19, each cooling body 23 having a diameter of 200 mm at the portion thereof covered with the heat

20 insulating member 27 was lowered into the molten aluminum 30. The body 23 was rotated at 400 r.p.m. while supply-ing a cooling fluid into its interior from the pipe 26. During operation, the molten aluminum was continuously supplied from the furnace 11 to the vessel 14 and tanks

25 15 to 18 so as to fill up these containers to a constant

level. Boron was also continuously placed into the vessel

14 to maintain the boron concentration at 0.008 wt. % at

all times. The eutectic impurity concentrations C1, C2,

C3 and C4 of the melt portions in the tanks 15 to 18

5   remained in the relation of C1   C2   C3   C4 from the

start of the operation, and a steady state was established

after the amount of aluminum deposit on the body 23 reached

a specified amount. When the effective distribution

coefficient of iron was 0.1 at a recovery ratio of 0.7,

10   the ratios of the iron concentrations C1, C2, C3 and C4 to

the iron concentration C0 of the molten aluminum supplied

from the furnace 11, namely C1/C0, C2/C0, C3/C0 and C4/C0,

levelled off at 1.3, 1.6, 2 and 2.7, respectively. The

operation was continued in this state. When checked for

15   impurity concentration, the resulting cast aluminum blocks

were found to contain 0.008 wt. % of Fe, 0.008 wt. % of

Si, 0.0001 wt. % of Ti, 0.0002 wt. % of V and 0.0006 wt.

% of B on the average.

Example 4

20   The operation of Example 3 was repeated under

the same conditions except that the recovery ratio was 0.4.

The operation was continued in a steady state. All the

aluminum blocks obtained were found to have average

impurity concentrations of: 0.005 wt. % Fe, 0.006 wt. %

25   Si, 0.0001 wt. % Ti, 0.0002 wt. % V and 0.0005 wt. % B.

Example 5

The operation of Example 3 was repeated under the same conditions as in Example 3 except that eight purifying tanks were used in place of the four tanks.

5 The operation was continued in a steady state. The aluminum blocks obtained were found to have the following average impurity concentrations: 0.0065 wt. % Fe, 0.007 wt. % Si, 0.0001 wt. % Ti, 0.0002 wt. % V, 0.0005 wt. % B. The aluminum had a higher purity than that obtained in

10 Example 3 with use of the four purifying tanks and was produced at twice the speed therein.

The present invention may be embodied differently without departing from the spirit and basic features of the invention. Accordingly the embodiments herein

15 disclosed are given for illustrative purposes only and are not in any way limitative. It is to be understood that the scope of the invention is defined by the appended claims rather than by the specification and that various alterations and modifications within the definition and

20 scope of the claims are included in the claims.

Claims

1. A process for producing high-purity aluminum comprising the steps of melting the aluminum to be purified and containing both eutectic impurities and peritectic impurities to obtain molten aluminum, and adding boron to the molten aluminum, characterised by rotating a cooling body (3, 23) as immersed in the boron-containing molten aluminum (1) while introducing a cooling fluid to the interior of the cooling body (3, 23) to crystallize high-purity aluminum on the surface of the body (3, 23)

2. A process according to claim 1, characterised by placing the molten aluminum into a holding container (2), adding the boron to the molten aluminum within the container (2), and rotating the cooling body (3) as immersed in the boron-containing molten aluminum within the container (2).

3. A process according to claim 1 or 2, characterised in that the molten aluminum is agitated after the boron is added thereto,

4. A process according to claim 1, characterised by placing the molten aluminum into a boron adding vessel (14), causing the molten aluminum placed into the vessel (14) to be forwarded to a plurality of purifying tanks (15-18) from tank to tank and to be discharged from the last tank (18), adding the boron to the molten aluminum within the vessel (14), and rotating one of the cooling bodies (23) as immersed in the boron-containing molten aluminum within each of the tanks (15-18).

5. A process according to claim 4, characterised in that the molten aluminum having the boron added thereto in the vessel is agitated.

6. A process according to claim 4 or 5, characterised in that the supply and the discharge of the molten aluminum are controlled so that the amounts of the molten aluminum in the purifying tanks (15-18) remain unchanged during operation, and the cooling bodies (23) are controlled so that equal amounts of high-purity aluminum are crystallized on the surfaces of the cooling bodies (23).

7. A process according to any of the preceding claims, characterised in that the speed of the relative movement between the cooling body (3, 23) and the molten aluminum is about 400 to about 8000 mm/sec when the cooling body is rotated.

FIG.3

24

26

19   27   30   19

13   25   13

15

28

12

FIG.1

5

4

2

1

3

FIG.2

FIG.4
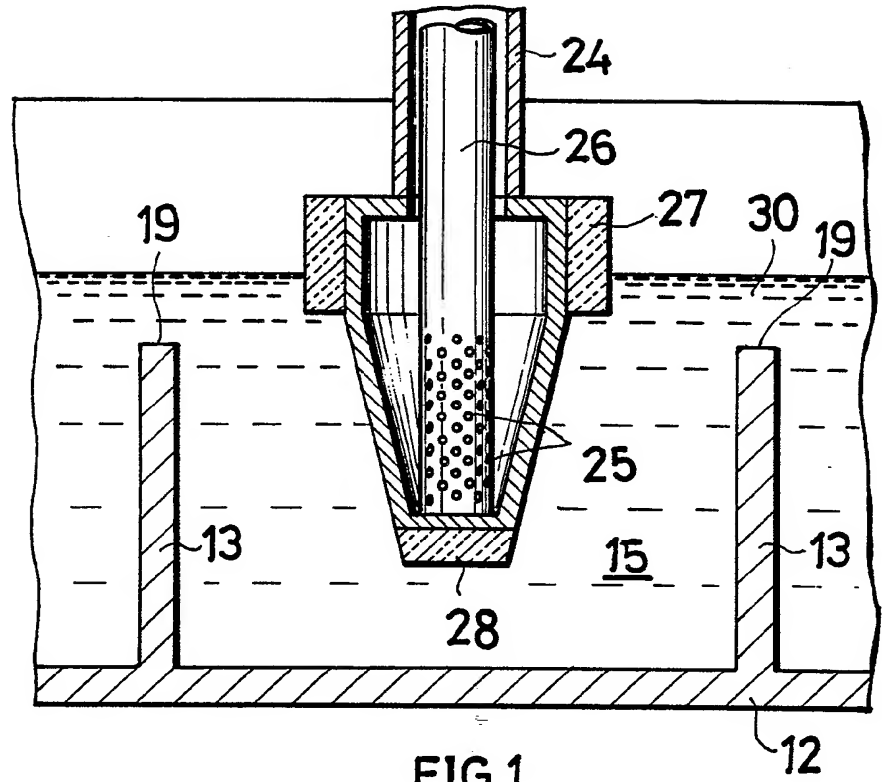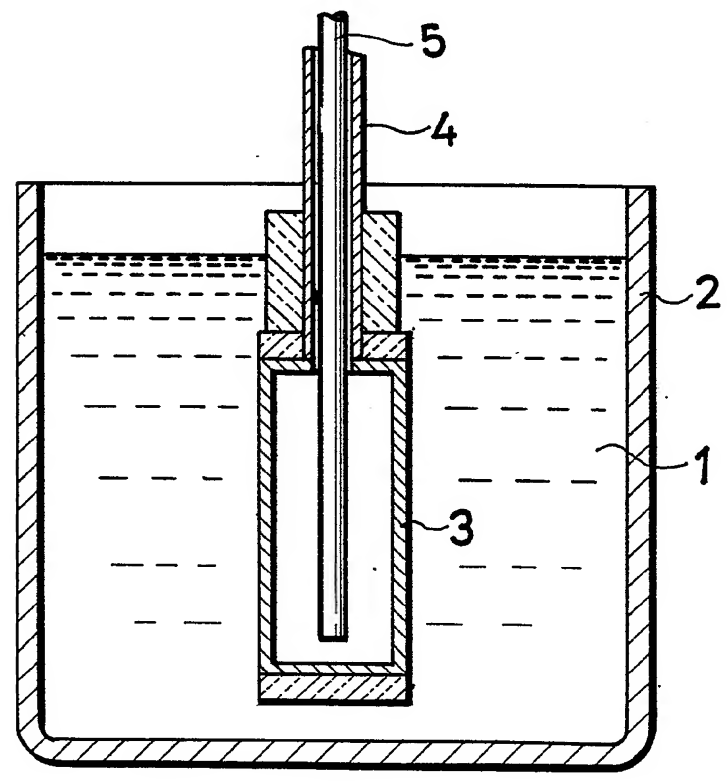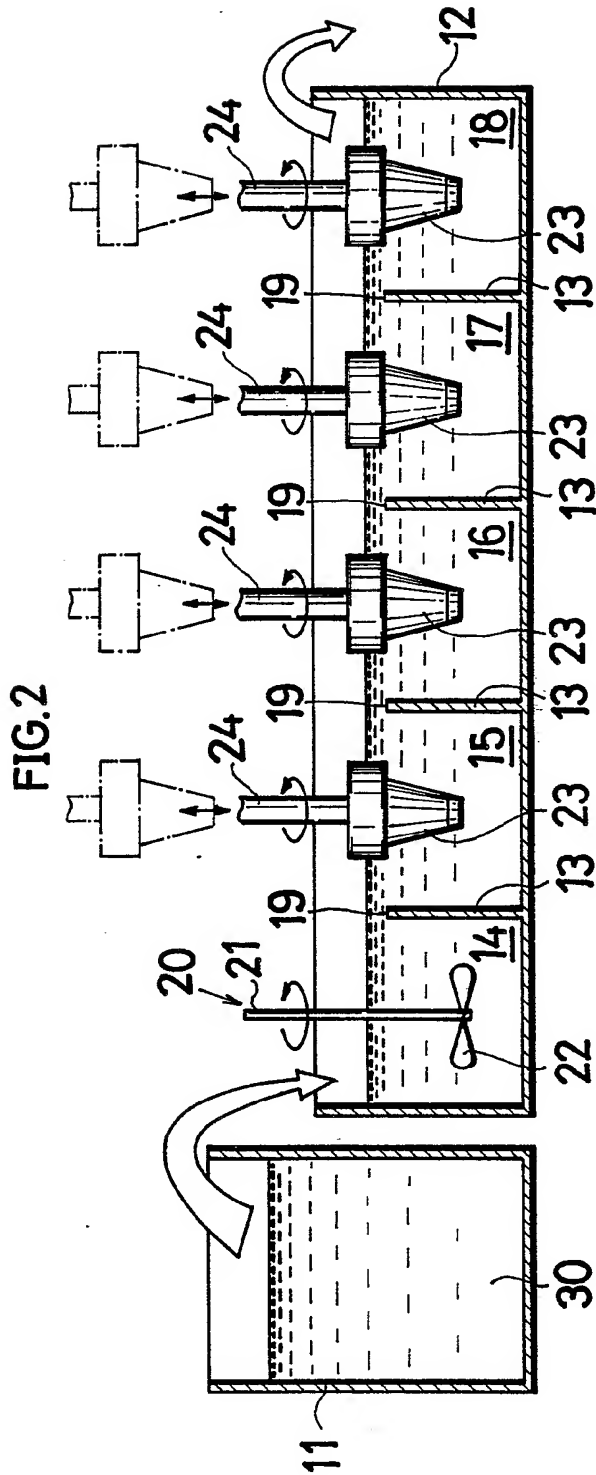
FIG.5
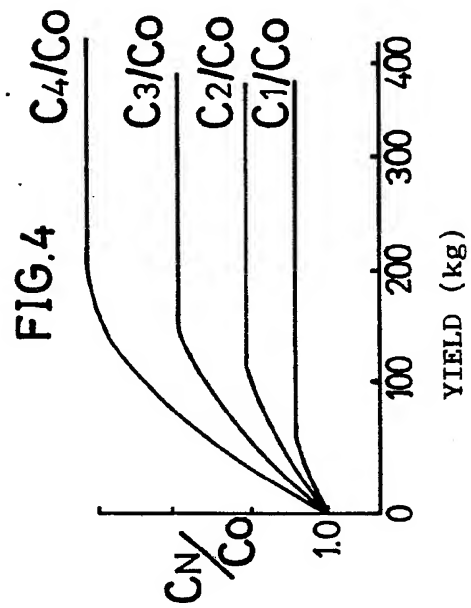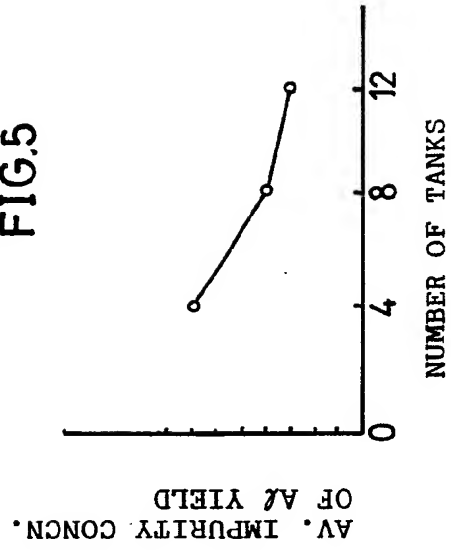
European Patent Office

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. ³)
Y	Patent Abstracts of Japan vol. 6, no. 3, 9 January 1982 & JP-A-56-127 735	1	C 22 B 21/06
Y	US-A-3 198 625 (P.T. STROUP) * Claim 1 *	1	
A,D	US-A-3 211 547 (N. JARRETT et al.)		
A,D	US-A-3 671 229 (M.E. FERBER et al.)		
A	US-A-2 471 899 (A. REGNER)		
A	US-A-3 239 899 (A.F. JOHNSON)		

TECHNICAL FIELDS SEARCHED (Int. Cl. ³)

C 22 B 21/06
C 22 B 9/02

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
BERLIN	24-03-1983	SUTOR W